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The Development of A Novel SR-CT Technique-Originated Equipment for Microwave Sintering

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Abstract

Microwave sintering has been paid a considerable amount of attention by researches as a novel technology for the preparation of dense structural ceramics recently. However, the mature theory has not been established due to the technical difficulties. Synchrotron Radiation X-ray Computed Tomography (SR-CT) technique is a latest non-destructive detection technology. Applying the SR-CT technology on the research of microwave sintering can realize the observation of the evolution of microstructure under microwave and high-temperature field in a non-destructive, 3D and real-time way, provide more accurate experiment data for revealing the kinetics mechanism, and offer direct foundation for establishing the theory of microwave sintering. But due to the high requirements of experiment skills, it is difficult to apply the SR-CT technique into the in-situ observation of the microstructure evolution process during microwave sintering. Especially, duo to the restriction of SR-CT experiment platform, to design a microwave sintering equipment is quite a big problem. In this paper, according to the analysis of the requirements of SR-CT technique and the restriction of SR-CT experiment platform, the corresponding solutions for these difficulties such as separated structure arrangement, two-ply heat insulation and high precise rotation device were put forward, and a microwave sintering furnace exclusively to SR-CT experiment was designed. The testing on the heat insulation structure and the precision calibration of the rotation device was conducted, the results show that the heat insulation effect is good and the rotation precise achieves the required standard. Besides, depending on this equipment, the first observation of the microstructure evolution of SiC during microwave sintering was carried out, and a few sintering phenomena during three sintering stages were clearly observed. The experiment results indicate that the all the specifications of the equipment meet the design requirements.

Keyword: Microwave sintering; synchrotron radiation X-ray computed tomography; microstructure evolution

1 Introduction

Microwave sintering is being developed as a novel technology for the preparation of dense structural ceramics recently [1,2], showing densification processes enhancement, less sintering time and decreasing grains size of

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products compared with conventional sintering [3,4]. However, the mature theory has not been established due to the technical difficulties. To find an appropriate experimental technique to observe the microstructure evolution process during microwave sintering in situ, comprehend the sintering phenomena and reveal the microwave sintering mechanism will provide more accurate experiment data for optimizing the microwave sintering technique and establishing the theory of microwave sintering. There are some optical and electron microscopy techniques [5] generally applied to investigate the microstructure of ceramic materials such as high resolution transmission electron microscope (HR-TEM) and scanning electron microscopy (SEM), which can but acquire high resolution images of the microstructure of the internal faults and supper-thin slices. Not only do these techniques destroy the original microstructure, but fails to observe the characteristics of microstructure evolution. Therefore, it is tremendously helpful to find an appropriate experiment method to powerfully support the mechanism revelation and process optimization of microwave sintering.

The Synchrotron Radiation X-ray Computed Tomography (SR-CT) technique is a latest non- destructive detection technology [6,7,8,9]. Via this technique, in situ observation of microstructure evolution of materials under extreme conditions (e.g., high pressure, high temperature, intense radiation, etc.) becomes possible. The SR-CT technology applied to the research of microwave sintering can realize the observation of the evolution of microstructure under microwave and high-temperature field in a non-destructive, 3D and real-time way, provide more accurate experiment data for revealing the kinetics mechanism, and offer direct foundation for establishing the theory of microwave sintering.

Currently, few scholars have tried to carry out the in-situ investigation of conventional sintering by the SR-CT technique. O. Lame [10], A. Vagnon [11], R.M-Atanasio [12], et al. have adopted this technique to observe the conventional sintering of metallic and ceramic materials. In our research group, we have also carried out the study on the conventional sintering of various ceramic materials [13,14]. Therefore, we are convinced that the SR-CT technique will provide the possibility of in-situ investigation on microwave sintering. However, due to the high requirement of experimental skills, it is difficult to apply the SR-CT technique into the in-situ observation of the microstructure evolution process during microwave sintering. Especially, duo to the restriction of the SR-CT experiment platform, to design a microwave sintering equipment is quite a big problem (such as structure arrangement, the requirement of X-ray passing, high precision rotation device, heat insulation and microwave leak).

In this paper, according to the analysis of the SR-CT technique requirements and the SR-CT experiment platform restriction, the corresponding solutions for these difficulties were put forward, and a microwave sintering furnace exclusively to SR-CT experiment was designed. The testing on the heat insulation device and the precision calibration on the rotation device has been carried out. Besides, the first observation of the microstructure evolution during microwave sintering on SiC was conducted on this equipment. The test and experiment results indicate that all the specifications of the equipment meet the design requirements, and it is feasible to apply the SR-CT technique on the study of microwave sintering.

2 The application exploration of the SR-CT technique on microwave sintering

2.1 The principle of SR-CT technique

The SR-CT technique is a non-destructive testing method by which the specimen passed through by synchrotron radiation X-ray is placed on a rotation device and the projection images of the specimen are received by an X-ray charge-coupled device (CCD). One projection image is collected each time when the specimen turns for an angle. After obtaining a set of projection data, reconstruction algorithm is used to obtain the internal microstructure of the sectional images. The 3-D images of the microstructure can be obtained from a series of sectional images. Reconstruction algorithms applied in the SR-CT technique are mainly filtered by back projection and iterative algorithms. Taking the limited time into account, filtered back projection algorithm is employed in this paper.

2.2 The specifications of the equipment and the technique difficulties

According to the principle of the SR-CT technique, the requirements of high-temperature microwave sintering and the restriction of the SR-CT experiment platform, we believe that the SR-CT microwave sintering furnace should consist of these parties: microwave source, microwave power control systems, microwave cavity, holes for x-ray passing and temperature measurement, heat insulation device, rotation device, etc. The working procedure of SR-CT analysis system that depends on the core equipment of SR-CT microwave sintering furnace is: a).Feed the microwave into the microwave cavity to establish the microwave heating field. b).The microwave heating field heats the specimen, and the microwave power control systems regulate the output power according to the specimen's temperature measured by the thermometer to realize the heating and heat insulation process. c).Let the specimen rotate the same angle each time and adopt the SR-CT technique to obtain the 3-D structure morphology of the specimen during the whole sintering process, then, the in-situ observation of the entire sintering process will be realized. The schematic diagram of SR-CT analysis system is shown in figure 1.

Based on the analysis above, the specifications of the equipment are listed as follows:

1).The maximal microwave power is set as 3KW, the power could be regulated continuously from 0 to 3KW, and the frequency of microwave is 2450±50MHZ which is within the national industry microwave frequency bands.

2).The distance between the synchrotron radiation ray and the platform is fixed (400mm), to match the experiment platform, the distance from the holes (for x-ray passing) on the furnace cavity to the furnace base should be less than 400mm.

3).The 3-D reconstruction technique requires that the positioning error of the specimen should be less than the

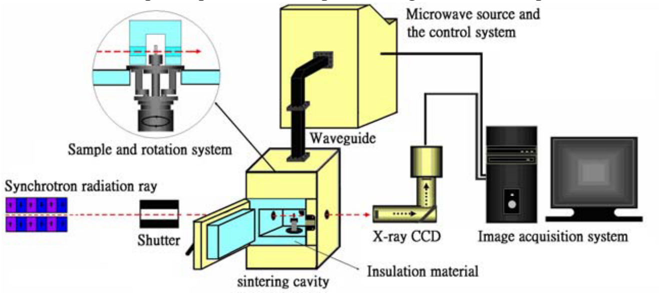


Figure 1 The schematic diagram of SR-CT analysis system of microwave sintering

size of the pixel resolution of CCD, but the resolution of human eye must be more than 3 or 4 times the size of the pixel resolution of CCD. In order to satisfy this requirement, the obliquity error of the axis of rotation of the rotation device should be less than 0.017°.

4).The upper measurement limitation of the thermometer should be more than 1500℃, the measurement error should be less than 1%. Here, the Reaytek TX-HT infrared thermometer is adopted, its temperature measurement scope ranges from 500℃ to 2000℃, the measurement error is ±1% or ±1.4 ℃ (choose the larger one).

These technical specifications are the necessary requirements that the microwave sintering equipment should meet. However, the combination of the SR-CT technique and microwave sintering technology calls for high experimental skills, and there are some crucial technical difficulties to be solved:

(1)Matching Requirement——the design of the SR-CT microwave sintering equipment should match the application environment. But: a).Holes on the furnace cavity are required for x-ray to passing. b).As describe in the section of specification requirements, the distance from the holes (on the furnace) to the furnace base should be less than 400mm. Therefore, how to design the structure and size of the equipment can realize the requirement of x-ray passing.

(2)Sintering Temperature Requirement——to achieve the high-temperature sintering, it is essential to design an effective heat insulation device. However, duo to: a).The size of the specimen is strictly limited by the SR-CT experiment platform (<φ5mm). Meanwhile, holes on the furnace cavity should be designed to ensure the x-ray pass through the specimen. b).The specimen is the only heat source and its heat could easily disperse into the surrounding space from its surface. So, how to design the heat insulation device can make the small specimen effectively sintered in the incomplete closed space.

(3)Rotation Device's working Environment and Precise Requirement —— in order to realize the specimen rotating at the range from 0 to 180° in the high-temperature microwave field, a special rotation device is required. But the ignition phenomenon can easily occur if the rotation device is placed in the microwave field. Besides, the rotation device will stop working unless the environment temperature is low than 200℃. So, how to design the rotation device can realize the high precise rotation of the specimen in the high temperature microwave field.

2.3 Design scheme of the SR-CT microwave sintering equipment

The difficulties discussed above are the key technical problems that need solution. In this paper, basing on the analysis of the technical difficulties, the separated structure design is put forward to realize the requirement of x-ray passing. As shown in figure 2. Furthermore, the design of two-ply heat insulation device and high precise rotation device are also been put forward to solve the problems of heat insulation and rotation, which will be described in details in section 2.1.

The separated-structure microwave sintering equipment is consisted of two parties: microwave source and sintering furnace. These two sections are connected by the waveguide which is used for microwave transmission. The assembly drawing and the size of each part are shown in Figure 3 (length unit: mm). The microwave source part is consisted of microwave generator, microwave power control systems, hardware protection systems, waveguide, circulators and water load. The sintering furnace part is consisted of heat insulation device, metal resonator, empty cavity (in the heat insulation structure), lifting base, furnace door, rotation system, heat gather cover and mechanical structure, etc. The holes are reserved on the right, left and back walls of the cavity for temperature measurement and X-ray passing. The infrared thermometer is fixed just out of the temperature-measuring hole to monitor the temperature of specimen during the sintering process. To prevent microwave leakage, metal wire cloth with fine holes is laid at the door gap (between the furnace door and door frame), and metal tubes (φ18mm×55mm) are fixed just out of the holes.

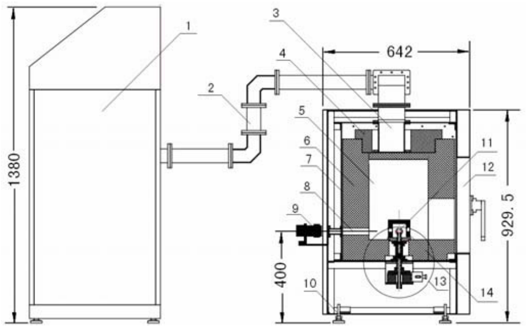


Figure.2 The assembly mechanical drawing of the SR-CT microwave sintering equipment
1,microwave source and the control systems; 2,waveguide; 3, flange; 4,6, polycrystalline mullite heating insulating material; 5, empty cavity; 7, metal parietal; 8,hole for temperature measurement; 9, Infrared Thermometer; 10, lifting base; 11, hole for x-ray passing; 12, furnace door; 13, rotation device; 14,heat insulation cover

3 Solutions to the key technical difficulties

As referred above, it is difficult to design the equipment duo to the requirements of heat insulation and rotation, etc. For these difficulties, the design of two-ply heat insulation structure and high precise rotation device are put forward, the design process is given as follows.

3.1 The design of two-ply heat insulation device

As we know, the sintering experiment is always carried out under high-temperature condition. But in our experiment, the specimen size is quite small, and there are some holes on the furnace cavity which will connect the internal space of the furnace with the external environment. So, it will be difficult to conduct the effective high-temperature sintering experiment if there is no good heat insulation structure. In order to solve this problem, the two-ply insulation structure is designed: the outer layer of insulation structure is to gather the microwave energy absorbed by the specimen, the inner insulation layer is for the further improvement of heat insulation, as well as inhibiting the heat exchange between the specimen and the external environment though the holes, the detailed design scheme is as follows:

The heat insulation material should have the properties of both microwave transparent and good heat insulation effect. Therefore, the polycrystalline mullite material is adopted. The outer heat insulation structure is designed as shown in figure 2 that marked with number 4 and 6. There are three holes (φ=20mm) on three sides of the cavity, two for x-ray passing and one for temperature measurement. Besides, there is a big hole (φ=85mm) at the bottom of the heat insulation structure to fix the revolving stage. On the top of revolving stage there is an insulation disc, which is used to prevent heat diffusion to ensure the rotation device work normally. There is an empty cavity inside of the heat insulation structure to place specimen. For easy operation, the empty cavity should not be too small.

If the specimen is directly heated in the empty cavity, only the inner part of the specimen will be effectively sintered, but the outer part will not. This case has been encountered when testing the heat insulation device. The preliminary analysis considers that the cause for this phenomenon maybe owing to the fact that the size of the specimen is too small and the output power of microwave is too low. However, it is useless when increasing the specimen size and enhancing the output of microwave power. After several tests and analysis, it is found that if only put a heat insulation cover on the specimen, the problem of incomplete sintering will be well solved. The heat insulation cover is an inverted -cylinder-like device, on the sides of which there are holes (φ=15mm) as well, as shown in figure 3. This design principle could be explained by the theory of heat transfer [15]: the reason why the specimen can't realize the entire sintering is that the microwave energy is selectively loading on the specimen, and there is no other heat source. So the heat can easily diffuse from the small specimen to the environment and it is difficult to realize entire sintering. However, after covering the heat insulation cover, the heat conduction distance dx in the heat conduction equation:

$$Q=-\lambda A dt/dx$$

will increase, as shown in figure 4. If the microwave power is invariable, when the system reaches the heat steady state, the conductive energy Q, the heat transfer area A and the thermal conductivity λ are invariant. So the value

$$dt=T_{specimen}-T_{environment}$$

will increase. Because the environment temperature will not change much, so the temperature of specimen $T_{specimen}$ will increase. Moreover, there are only three holes on the cover, so the thermal convection rate between the specimen and the external environment will reduce a lot, as well as the thermal convection area. So the surface heat transfer coefficient H and the convection area A will reduce a lot in the heat convection equation:

$$Q=HAdt=HA(T_{specimen}-T_{environment})$$

Similarly, the value of $T_{specimen}$ will increase, and the temperature of the specimen's surface will increase as well. Thereby, the specimen can realize the entire sintering.

The sintering experiment on SiC has been carried out on the equipment to test the improved heat insulation device. The temperature of the specimen can reach over 1500℃, and the specimen became a close-to-ideal dense

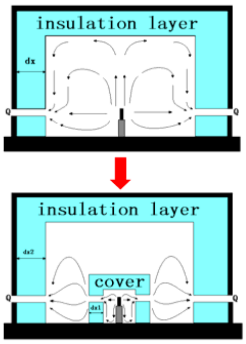


Figure 3 The design of two-ply heat insulation structure

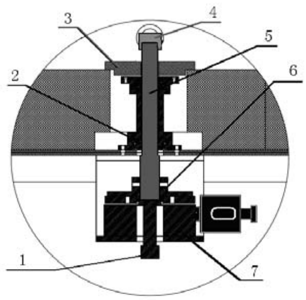


Figure.4 The rotation device

1,metal bolt; 2,position limition device; 3,polycrystalline mullite disc; 4,crucible; 5,cylindrical corundum; 6, metal coupling; 7,MRS revolving stage

body after microwave sintering, the result shows that the design of heat insulation structure is efficacious.

3.2 The design of rotation device

The rotation device is one of the essential structures for microwave sintering equipment, and its working precise will probably affect the quality of the construction images. In order to ensure the high precise operation and the low working temperature (lower than 200℃) for the rotation device, the rotation device design scheme is: fix the revolving stage under the resonator cavity, connect it with the resonator cavity by the cylindrical corundum which is used to transfer the rotation angle from the revolving stage to the specimen. A position limiting device was fixed out of the cylindrical corundum to enhance the operation precise of the rotation device. The detailed design scheme is as follows:

In order to transfer the rotation angle synchronously from the revolving stage to the specimen, and make sure the revolving stage work under the tolerable temperature, set the revolving stage 100mm under the cavity, connect it with the cylindrical corundum by a metal shaft coupling which has the effect of cooling as well. Put the top of the cylindrical corundum into the cavity to place the specimen, let the specimen rotate synchronously with the cylindrical corundum during the experiment. As shown in figure 4.

In order to increase the coaxiality of cylindrical corundum, the position limit device is designed, as shown in figure 4 marked with number 2. The position limit device is a hollow metallic sheath, the two ends of which are ball bearings. The assemblage between the inner circumference of metallic sheath and the outer circumference of cylindrical corundum satisfies clearance fit, the inner circumference of ball bearing and the outer circumference of cylindrical corundum satisfies transition fit. When assembling the position limit device, put the ball bearing out of the cylindrical corundum first, and fix the position limit device to the bottom of the cavity (fixed position can be regulated). The position limit device can make the cylindrical corundum rotate just within the inner ring of the ball bearing, which can improve the coaxiality of cylindrical corundum quite a lot. Under the cylindrical corundum there is a metal screw bolt, the height adjustment of the cylindrical corundum can be achieved by regulating the height of this screw bolt, so that the specimen can be exactly passed though by the synchrotron radiation x-ray.

4 The precision calibration of rotation device

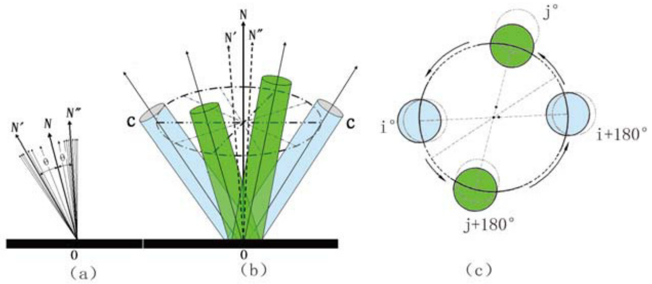


Figure.5 The diagrammatic sketch of the obliquity of cylindrical corundum

On the principle of CT technique, the reconstruction image quality is influenced by the spatial resolution of the x-ray, x-ray source noise, x-ray CCD array distribution accuracy and the position accuracy of specimen, and the position accuracy of specimen is determined by the precise of rotation device. As far as the equipment is concerned, just the position accuracy of cylindrical corundum is considered. Ideally, the cylindrical corundum should rotate around the same rotation axis, but duo to the limitation of machining precision and the vibration of the revolving stage, the rotation axis of the cylindrical corundum will vibrate in a certain range. As shown in figure 5(b), ON is the ideal rotation axis, ON' and ON" are two of the actual rotation axis, and there is an angle difference θ between ON and ON' or ON". Thence, on the top of the cylindrical corundum, the angle difference θ will bring the distance error

$$\triangle L = R\theta,$$

R is the length from the top of the cylindrical corundum to point O. According to the CT principle, if the ΔL is less than the size of the CCD pixel, the reconstructed image will be accurate. In the experiment, the size of CCD pixel is 7μm, and the length of R is 100mm. Based on the analysis above, we can infer that:

$$\triangle L=R\theta<7\mu m$$

So,

$$\theta<7\times10^{-5}\ \text{rad},$$

However, the resolution of human eye should be more than 3 or 4 times of the size of the pixel resolution of CCD. In addition, the cylindricity of the cylindrical corundum is not ideal, these factors will bring measurement error.

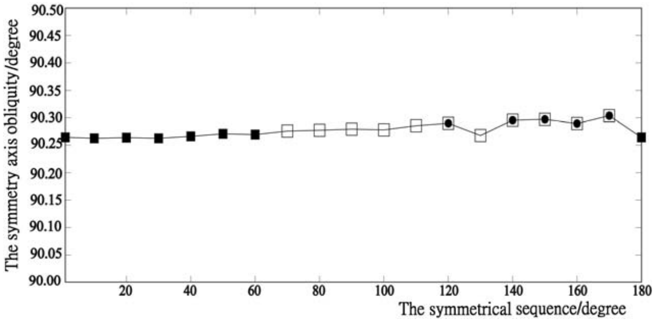


Figure.6 The symmetrical axis obliquity distribution of cylindrical corundum

Ignoring these factors, the rotation precise can be set as: θ<3.0×10-4 rad (that is 0.017°).

In the calibration process, the position of i° and i+180° are considered as a couple of symmetrical position, each symmetrical position will correspond a rotation axis, as shown in figure 5(c) (this figure is corresponding with the C-C cross-section in figure 5(b), the colored circles represent the ideal position of the cylindrical corundum during rotating, and the dotted line circles represent the real position of the cylindrical corundum). The obliquity distribution of cylindrical corundum will vibrate in a certain range, as shown in figure 5(a). During the calibration process, let the cylindrical corundum run a lap, use the optical method to obtain the symmetry axis obliquity distribution of cylindrical corundum. The statistical data is presented visually in figure 6.

From figure 6, it is found that the point at the 130° symmetrical position is a singular one which should be omitted. The left obliquity distribution has two intensive regions: points marked as ■ and ◙ respectively correspond with two intensive regions, and points marked as □ are the obliquities that distribute between the two intensive regions. In the intensive region, the obliquities vibrate in a small range, which must be duo to the vibration of rotation device and the random perturbation from the external environment. Without consider such interference, the obliquities of the rotation axis in each intensive region should be approximately equal to the average value of each intensive region, as the ON' and ON" shown in figure 5(a). Based on the analysis above, we can obtain the results as shown in table-1.

Table 1 the calibration results of rotation device

Parameters	Physical meaning	Value
Ψ_{max}	The maximal obliquity of the rotation axle	90.2966°
Ψ_{min}	The minimum obliquity of the rotation axle	90.2655°
R	The distance from the top if the cylindrical corundum to the bottom of the rotation axle	100mm
$\theta=(\Psi_{max}-\Psi_{min})/2$	The maximal difference of rotation axle	2.71×10^{-4} rad
$\triangle L=R\theta$	The maximal distance error at the top of the cylindrical corundum	27.14μm

According to the calibration results in table-1, the maximal difference of the obliquity of rotation axis θ=2.71×10-4 rad<3.0×10-4 rad , which meets the required precise.

5 Experiment

The experiment was performed at the BL13W1 beamline in Shanghai Synchrotron Radiation Facility (SSRF). The real picture of the SR-CT microwave sintering equipment is shown in figure 7, and the microwave sintering experiment on SiC specimen was carried out on this equipment. In the experiment, the SiC particles were poured into the quartz tube (φ=2.5mm) which were vertically set in the crucible. The output power is 3KW, and the sintering temperature is 1500℃, sintering time is one hour. During the sintering process, the SR-CT technique was

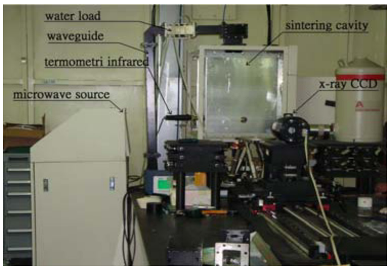
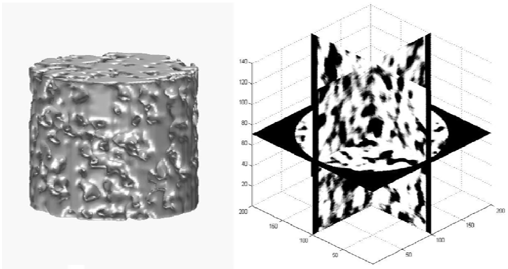


Figure.7 The real picture of SR-CT microwave sintering equipment



(a) (b)

Figure.8 The 3-D microstructure morphology of SiC

adopted to investigate the microstructure evolution process of SiC specimen. Figure 8(a) is the three-dimensional microstructure morphology of the specimen at a certain sintering time. From this figure, sintering phenomena during three sintering stages, including grain contact, grain growth, sintering neck growth, and pore spheroidization were clearly observed. Figure 8(b) is the two-dimensional slices images in the three orthotropic planes (white represents particle, black represents pore). Depending on these slices images, the parameters such as sintering neck size, porosity, grain size, relative density can be obtained by the digital image processing method, which will provide accurate experimental data for the further theoretical analysis and modeling establishment. The experiment results indicate that all the specifications of the equipment meet the design requirements, and it is feasible to apply the SR-CT technique on the study of microwave sintering.

6 conclusion

The SR-CT technique-originated equipment for microwave sintering was designed depending on the principle of SR-CT technique and high-temperature microwave sintering technique, the analysis of the working environment of experiment platform. The SR-CT technique was firstly applied in the study of microwave sintering. The design of separated structure, two-ply heat insulation device and high precise rotation device resolves the technical problems of x-ray passing, heat insulation, and specimen rotating respectively. The two-ply heat insulation device realizes the high-temperature sintering for small size specimen in an incompletely closed space. In the test of the heat insulation device, SiC specimen was used for microwave sintering, the sintering temperature reached over 1500℃, and the SiC specimen became a close-to-ideal dense body after microwave sintering. The rotation device realizes the high precise rotation of the specimen in high-temperature microwave field, the precision calibration results show that the obliquity deviation of the axis of rotation of cylindrical corundum is 2.71×10-4 rad, which meets the required precision. Basing on this equipment, the first observation of microstructure evolution during microwave sintering on SiC specimen was carried out. The experiment results indicate that all the specifications of the equipment meet the design requirements. The successful design of this equipment will offer powerful experimental support for revealing the kinetics mechanism and establishing the theory of microwave sintering.

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